

Claims.

1. A process for demineralizing coal comprising:

- (a) forming a slurry of coal particles in an alkali solution,
- 5 (b) maintaining the slurry at a temperature of 150-250°C under a pressure sufficient to prevent boiling;
- (c) separating the slurry into an alkalized coal and a spent alkali leachant;
- 10 (d) forming an acidified slurry of the alkalized coal, said acidified slurry having a pH of 0.5-1.5;
- (e) separating the acidified slurry into a coal-containing fraction and a substantially liquid fraction;
- 15 (f) subjecting the coal-containing fraction to a washing step in which the coal-containing fraction is mixed with water and a polar organic solvent or water and an organic acid to form a mixture; and
- (g) separating the coal from the mixture in step (f).

2. A process as claimed in claim 1 wherein the coal provided to step (a) is sized such that 100% is less than 1mm.

3. A process as claimed in claim 2 wherein the coal provided to step (a) is sized such that 100% less than 0.5mm.

4. A process as claimed in claim 2 or claim 3 wherein the coal provided to step (a) contains 5% by weight smaller than 20 microns.

5. A process as claimed in any one of the preceding claims wherein the slurry formed in step (a) has a coal concentration of from 10% to 30% by weight.

6. A process as claimed in claim 5 wherein the coal concentration in the slurry is about 25% by weight.

7. A process as claimed in any one of the preceding claims wherein an alkali concentration in a liquid phase of the slurry is in the range of 8% to 20% by weight (calculated as NaOH equivalent).

8. A process as claimed in claim 7 wherein the alkali concentration is from 13% to 15% by weight (calculated as NaOH equivalent).

9. A process as claimed in any one of the preceding claims wherein the slurry is heated to a temperature of from 220-250°C in step (b).

10. A process as claimed in any one of the preceding claims wherein the slurry is maintained at an elevated temperature in step (b) for a period of from 15 to 60 minutes.

11. A process as claimed in any one of the preceding claims wherein a rate of heating the slurry is maintained at a rate of less than 2°C per minute in the temperature range of 150°C to 250°C.

12. A process as claimed in any one of the preceding claims wherein the slurry in step (b) is maintained at the autogenous pressure of the heated slurry to prevent the slurry from boiling.

13. A process as claimed in any one of the preceding claims wherein step (c) takes place at a temperature of from 30°C to 80°C.

14. A process as claimed in claim 13 wherein the slurry from step (b) is cooled to a temperature of from 30-80 C at a cooling rate of less than 20°C/minute and at 2 C per minute whilst the temperature of the slurry is in the range of 240°C - 150°C.

15 A process as claimed in any one of the preceding claims wherein the alkalized coal recovered from step (c) is washed to remove excess alkali.

5 16. A process as claimed in any one of the preceding claims wherein the alkalized coal from step (c) is treated to remove sodium aluminosilicates therefrom prior to sending to step (d).

10 17 A process as claimed in any one of the preceding claims wherein step (d) comprises mixing the coal from step (c) with water or an acid solution to obtain a slurry having a coal concentration that falls within the range of 5% to 20% by weight.

18. A process as claimed in claim 17 wherein the slurry has a coal concentration of about 10% by weight.

15 19. A process as claimed in any one of the preceding claims wherein the slurry in step (d) contains a mineral acid.

20 20. A process as claimed in claim 19 wherein the mineral acid is sulphuric acid or hydrochloric acid.

21. A process as claimed in any one of the preceding claims wherein the slurry of step (d) has a pH that falls in the range of 0.5 to 1.5.

25 22. A process as claimed in claim 21 wherein the pH of the slurry is about 1.0.

23. A process as claimed in any one of the preceding claims wherein the temperature of the slurry in step (d) falls within the range from 20°C to 90°C.

30 24. A process as claimed in claim 23 wherein the temperature falls within the range of from 30°C to 60°C.

25. A process as claimed in any one of the preceding claims wherein the coal is maintained in contact with the acid solution in step (d) for a period of at least 1 minute.

26. A process as claimed in claim 25 wherein the coal is maintained in contact with the acid solution in step (d) for a period of about 60 minutes.

27. A process as claimed in any one of the preceding claims wherein the coal fraction from step (e) is re-slurried with water and acid and brought to a pH of between 0.5 and 1.0 for a further period of time of greater than 1 minute

28. A process as claimed in claim 27 wherein the step of re-slurrying the coal is repeated between one and four times.

29. A process as claimed in any one of the preceding claims wherein step (f) comprises mixing the coal-containing fraction with a solution of water and an alcohol selected from ethanol, methanol, propanol or mixtures thereof.

30. A process as claimed in claim 29 wherein the organic solvent is ethanol.

31. A process as claimed in any one of the preceding claims wherein, in step (f), the coal is mixed with water and polar organic solvent such that a slurry having a solids content of 10-30% by weight is formed.

32. A process as claimed in claim 31 wherein the slurry has a pH of from 1.5 to 2.5.

33. A process as claimed in any one of claims 29 to 32 wherein the slurry heated to a temperature of from 240°C to 280°C in step (f).

34. A process as claimed in claim 33 wherein the slurry is kept at elevated temperature for a period of between 1 minute and 60 minutes.

35. A process as claimed in claim 33 wherein the slurry of coal/water/polar organic solvent is heated at a heating rate of between 2°C per minute and 20°C per minute.

36. A process as claimed in any one of claims 1 to 28 wherein step (f) comprises
5 subjecting the coal-containing fraction to a hydrothermal washing step in which the coal-containing fraction is mixed with water and an organic acid selected from citric acid, chloroacetic acid, malonic acid, malic acid or mixtures thereof.

37. A process as claimed in claim 36 wherein the organic acid is citric acid and a citric
10 acid solution containing between 5% and 20% by weight citric acid (hydrated basis) is added to the coal-containing fraction.

38. A process as claimed in claim 37 wherein the slurry is heated to a temperature of
15 between 240°C to 280°C.

39. A process as claimed in claim 37 wherein slurry is heated to a temperature of
20 between 150°C and 160°C.

40. A process as claimed in claim 38 or 39 wherein the pressure is maintained at a level
25 sufficient to prevent boiling.

41. A process as claimed in any one of claims 38 to 40 wherein the slurry is at elevated
30 temperature for a period of between 1 minutes and 60 minutes.

42. A process as claimed in any one of claims 38 to 41 wherein the slurry is heated to the
35 elevated temperature at a heating rate of between 2°C per minute and 20°C per minute.

43. A process as claimed in any one of the preceding claims the coal recovered from step
40 (g) is washed with water.

44. A process as claimed in any one of the preceding claims wherein demineralised coal recovered from step (g) has an ash content of from 0.01-0.2%, by weight.

45. A process for demineralising coal comprising the steps of alkali digestion followed by acid soaking and wherein coal from the acid soaking step is subjected to a washing step in which the coal-containing fraction is mixed with water and a polar organic solvent or water and an organic acid to form a mixture, and separating the coal from the mixture.

46. A process for demineralizing coal comprising:

- (a) forming a slurry of coal particles in an alkali solution, said slurry containing 10-30% by weight coal;
- (b) maintaining the slurry at a temperature of 150-250°C under a pressure sufficient to prevent boiling;
- (c) separating the slurry into an alkalized coal and a spent alkali leachant;
- (d) forming an acidified slurry of the alkalized coal, said acidified slurry having a pH of 0.5-1.5; and
- (e) separating the acidified slurry into a coal-containing fraction and a substantially liquid fraction.

47. A process as claimed in claim 29 wherein the temperature used in step (f) is from 10 C to ambient temperature.

48. A process as claimed in any one of claims 1 to 44 wherein the spent alkali leachant from step (c) is treated to regenerate caustic and to recover minerals.

49. A process as claimed in claim 48 wherein the spent alkali leachant is treated by mixing with one or more of calcium oxide, calcium hydroxide, magnesium oxide, magnesium hydroxide, or mixed oxides or hydroxide of calcium and magnesium derived

from dolomite to precipitate soluble silicate and aluminate ions and from soluble sodium hydroxide.

50. A process as claimed in any one of claims 1 to 44 or 48 to 49 wherein the
5 substantially liquid fraction of step (e) is treated to regenerate a caustic solution and to recover minerals.

51. A process as claimed in claim 50 wherein the substantially liquid fraction is mixed
with one or more of calcium oxide, calcium hydroxide, magnesium oxide, magnesium
10 hydroxide, or mixed oxides or hydroxide of calcium and magnesium derived from dolomite.